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(54) METHOD FOR HEAT-TREATING NANO-CRYSTALLINE ALLOY

(57)Abstract:

PURPOSE: To produce a nano-crystalline alloy excellent in magnetic properties by subjecting an amorphous alloy to heat treatment in a gaseous atmosphere having a specified dew point, crystallizing the same and allowing crystal grains having a specified average grain size to occupy a part of the structure.

CONSTITUTION: An amorphous alloy is prep'd. by a superrapid cooling method such as a single roll method, which is subjected to heat treatment and is crystallized to produce a nano-crystalline alloy in which crystal grains having 430nm average grain size occupy at least a part of the structure. At this time, it is subjected to heat treatment (at about 550°C) in an atmosphere of gaseous argon, gaseous helium, gaseous nitrogen or a gaseous mixture thereof. Thus, the nano- crystalline alloy having an ultrafine grain structure and excellent in magnetic properties can be obtd.

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(54)【発明の名称】 ナノ結晶合金の熱処理方法

(57)【要約】

【目的】 超微細な結晶粒組織を有する磁気特性に優れたナノ結晶合金の熱処理方法を提供することである。

【構成】 アモルファス合金を結晶化させて平均結晶粒径が30nm以下である結晶粒が組織の少なくとも一部を占めるナノ結晶合金を露点が-30°C以下のガス雰囲気中で熱処理を行なう。

【特許請求の範囲】

【請求項1】 アモルファス合金を結晶化させて平均結晶粒径が30nm以下である結晶粒が組織の少なくとも一部を占めるナノ結晶合金を製造する熱処理方法において、前記熱処理を露点が-30°C以下のガス雰囲気中で行なうことを特徴とするナノ結晶合金の熱処理方法。

【請求項2】 前記露点が-60°C以下のガス雰囲気中であることを特徴とする請求項1に記載のナノ結晶合金の熱処理方法。

【請求項3】 前記雰囲気ガスがアルゴンガス、ヘリウムガス、窒素ガスあるいはこれらの混合ガスであることを特徴とする請求項1または請求項2に記載のナノ結晶合金の熱処理方法。

【請求項4】 ナノ結晶合金が

一般式: $(Fe_{1-x}M_x)_{100-x-y-z-b}A_xM'_yM''_zX_b$ (原子%)で表され、式中MはCo,Niから選ばれた少なくとも1種の元素を、AはCu,Auから選ばれた少なくとも1種の元素、M'はTi,V,Zr,Nb,Mo,Hf,TaおよびWから選ばれた少なくとも1種の元素、M''はCr,Mn,Al,Sn,Zn,Ag,In,白金属元素,Mg,Ca,Sr,Y,希土類元素,N,OおよびSから選ばれた少なくとも1種の元素、XはB,Si,C,Ge,GaおよびPから選ばれた少なくとも1種の元素を示し、a,x,y,zおよびbはそれぞれ0≤a≤0.5、0≤x≤10、0.1≤y≤20、0≤z≤2、0.2≤b≤30を満足する数で表される組成であることを特徴とする請求項1乃至請求項3のいずれかの項に記載のナノ結晶合金の熱処理方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】 本発明は、超微細な結晶粒組織を有する磁性部品等に使用されるナノ結晶合金の熱処理方法に関する。

【0002】

【従来の技術】 ナノ結晶合金は優れた軟磁気特性を示すため、コモンモードチョークコイル、高周波トランス、漏電警報器、パルストラ ns等の磁心に使用されている。代表的組成系は特公平4-4393号公報や特開平1-242755号公報に記載の合金系等が知られている。これらのナノ結晶合金は、通常液相や気相から急冷し非晶質合金とした後、これを熱処理により微結晶化することにより作製されている。液相から急冷し、非晶質合金とする方法としては単ロール法、双ロール法、遠心急冷法、回転液中紡糸法、アトマイズ法やキャビテーション法等が知られている。また、気相から急冷し、非晶質合金とする方法としては、スパッタ法、蒸着法、イオンプレーティング法等が知られている。ナノ結晶合金はこれらの方法により作製した非晶質合金を微結晶化したもので、非晶質合金にみられるような熱的不安定性がほとんどなく、高飽和磁束密度、低磁歪で優れた軟磁気特性を示すことが知られている。更にナノ結晶合金は経時変化が小さく、温度特性にも優れていることが知られている。

【0003】

【発明が解決しようとする課題】 ナノ結晶合金の熱処理は通常窒素ガスやアルゴン等の不活性ガス中で行われるが使用するガスによりしばしば磁気特性に劣化が生ずる場合があり問題となっていた。本発明の目的は均一で超微細な結晶粒組織を有する磁気特性に優れたナノ結晶合金の熱処理方法を提供することである。

【0004】

【課題を解決するための手段】 上記問題点を解決するために本発明者らは、鋭意検討を進めた結果、表面に酸化物や水酸化物等の変質層がある場合には、磁気特性が劣化したり、磁気特性自体のばらつきが大きいことを知見した。そこでこの変質層の発生を防ぐには、熱処理中のガス雰囲気中の水分量を制御する、つまりガス雰囲気中の露点を制御すれば良いことを知見し、本発明に想到した。すなわち、本発明は、アモルファス合金を結晶化させて平均結晶粒径が30nm以下である結晶粒が組織の少なくとも一部を占めるナノ結晶合金を製造する熱処理方法において、熱処理を露点が-30°C以下のガス雰囲気中で行なうことを特徴とするナノ結晶合金の熱処理方法である。露点を-30°C以下に限定したのは、露点が-30°Cを越えると合金表面変質層が増加し、透磁率等の磁気特性が劣化するためである。

【0005】 特に露点が-60°C以下のガス雰囲気中である場合は磁気特性が更に向上し、より好ましい結果が得られる。露点-30°Cは337.7mg/m³の水分量、露点-60°Cは10.93mg/m³の水分量に相当する。雰囲気ガスとして、不活性のアルゴンガス、ヘリウムガス、窒素ガスあるいはこれらの混合ガスを使用した場合は優れた磁気特性が得られ本発明の効果が顕著である。

【0006】 ナノ結晶軟磁性合金は特に

一般式: $(Fe_{1-x}M_x)_{100-x-y-z-b}A_xM'_yM''_zX_b$ (原子%)で表され、式中MはCo,Niから選ばれた少なくとも1種の元素を、AはCu,Auから選ばれた少なくとも1種の元素、M'はTi,V,Zr,Nb,Mo,Hf,TaおよびWから選ばれた少なくとも1種の元素、M''はCr,Mn,Al,Sn,Zn,Ag,In,白金属元素,Mg,Ca,Sr,Y,希土類元素,N,OおよびSから選ばれた少なくとも1種の元素、XはB,Si,C,Ge,GaおよびPから選ばれた少なくとも1種の元素を示し、a,x,y,zおよびbはそれぞれ0≤a≤0.5、0≤x≤10、0.1≤y≤20、0≤z≤2、0.2≤b≤30を満足する数で表される組成の場合に優れた軟磁気特性が得られる。

【0007】 前述の結晶は主にbccFe相であり、Siを含む場合はbcc相中にはSiが固溶し規則格子を含む場合もある。また、Si以外の元素たとえばB,Al,Ge,Zr,Ca等を固溶している場合もある。前記結晶相以外の残部は主にアモルファス相であるが、実質的に結晶相だけからなる場合もある。本発明に係わる合金は、前記組成のアモルファス合金を単ロール法等の超急冷法により作製後、これを磁心の形状に加工し、露点が-30°C以下のガス雰囲

気中で結晶化温度以上に昇温し熱処理を行い、平均粒径30nm以下の超微結晶粒を形成することにより作製する。熱処理の際磁場を印加し磁場中熱処理を行っても良い。

【0008】炉内の雰囲気ガスを強制的に移動させることは、磁心表面からの結晶化による発生する熱の放熱が良くなるため、磁心温度の異常な上昇を低く抑えることができるため、より好ましい結果を得ることができる。

炉外から炉内に雰囲気ガスを導入するとともに炉内のガスを排出し、炉内の雰囲気ガスを強制的に移動させることも同様な効果を得ることができる。炉内の雰囲気ガスをファン等で強制的に攪拌させ移動させることも磁心表面からの放熱を良くすることができるため同様な効果を得ることができる。

【0009】ナノ結晶合金表面温度と炉の設定温度の差が50°C以下になるように雰囲気ガスの炉内移動量を調整する機構を設けることにより、形状が大きくなつた場合にも容易に対応可能となる。特にナノ結晶合金表面温度と炉の設定温度の差が10°C以下である場合は特性の劣化および特性のばらつきが小さく好ましい結果を得られる。

【0010】

【作用】本発明において熱処理時に使用する雰囲気ガスの露点を-30°C以下とすることにより磁気特性に影響を与える合金表面変質層を少なくでき、磁気特性の劣下を減少させることができる。

【0011】

【実施例】以下本発明を実施例にしたがって説明するが本発明はこれらに限定されるものではない。

(実施例1) 原子%でCu 1%, Nb 3%, Si 15.4%, B 6.5%残部実質的にFeからなる合金溶湯を単ロール法により急冷し、幅5mm厚さ18μmのアモルファス合金を得た。このアモルファス合金を外径20mm、内径12mmに巻回し、トロイダル磁心を作製した。作製した磁心を表1に示す露点が異なる窒素ガス雰囲気の550°Cに保った熱処理炉に挿入し、30分保持後炉から取り出し空冷した。熱処理後の合金は粒径約12nmのbcc結晶が組織のほとんどを占めていた。

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合金は粒径約12nmのbcc結晶が組織のほとんどを占めていた。1kHzにおける比初透磁率 μ_r を表1に示す。表1から分るように露点が-30°C以下になると μ_r が著しく向上することが分る。特に-60°C以下で高い透磁率が得られた。

【0012】

【表1】

露点 (°C)	μ_r
-10	63200
-20	73000
-30	90100
-50	95500
-60	97200
-70	98300
-80	98500

【0013】(実施例2)表2に示す組成の合金溶湯を単ロール法により急冷し、幅25mm厚さ16μmのアモルファス合金を得た。このアモルファス合金を外径20mm、内径12mmに巻回し、トロイダル磁心を作製した。作製した各磁心をそれぞれ露点が-65°Cと-10°Cの表2に示す雰囲気ガスの550°Cに保った熱処理炉に挿入し30分保持後炉から取り出し空冷した。熱処理後の合金は粒径約12nmのbcc結晶が組織のほとんどを占めていた。得られた磁気特性を表2に示す。表2から分るように露点が-65°Cと低い方が透磁率が向上することが分る。更に同様の条件で長さ200mmの前記合金を熱処理し、1kHzにおける比透磁率 μ_r を測定した。測定後の試料表面をエッティングにより除去し、再度比透磁率 μ_{r0} を測定した。エッティング前の比透磁率とエッティング後の比透磁率の比 μ_{r0}/μ_r を表3に示す。

【0014】

【表2】

組成 (at%)	雰囲気ガス	μ_r [露点-65°C]	μ_r [露点-10°C]
Fe _{0.1} Cu ₁ Nb ₃ Si _{14.5} B _{0.2}	アルゴン	97200	62100
Fe _{0.1} Cu ₁ Nb ₃ Ti _{0.7} Si ₁₄ B ₀	窒素	98100	71500
Fe _{0.1} Cu ₁ Nb ₃ Si _{14.7} B ₂ Sn _{0.01}	ヘリウム	96200	74800
Fe _{0.1} Cu ₁ Nb ₃ Mo _{0.4} Si _{15.5} B _{0.2}	アルゴン	93800	73200
Fe _{0.1} Cu ₁ Nb ₃ Mn _{0.2} Si _{15.5} B _{0.5}	窒素	91200	71800
Fe _{0.1} Cu ₁ Nb ₃ Si _{15.5} B ₂ Ga _{0.3}	ヘリウム	89600	69500
Fe _{0.1} Cu ₁ Nb _{2.5} Cr _{0.5} Si _{13.5} B _{0.5}	窒素	88700	66800
Fe _{0.1} Cu ₁ Nb ₃ Si _{14.7} B ₂ Al _{0.01} Sn _{0.01}	窒素	91200	71900
Fe _{0.1} Cu ₁ Nb _{2.7} Mo _{0.5} Si ₁₅ B ₂ Co _{0.01}	窒素	87300	65900
Fe _{0.1} Cu _{1.5} Nb _{3.5} Si _{14.7} B ₂ Ge _{1.5}	窒素	91300	72300

【0015】

* * 【表3】

組成 (at%)	雰囲気ガス	μ_{re}/μ_r [露点-65°C]	μ_{re}/μ_r [露点-10°C]
Fe _{0.1} Cu ₁ Nb ₃ Si _{14.5} B _{0.2}	アルゴン	1.02	1.53
Fe _{0.1} Cu ₁ Nb ₃ Ti _{0.7} Si ₁₄ B ₀	窒素	1.09	1.36
Fe _{0.1} Cu ₁ Nb ₃ Si _{14.7} B ₂ Sn _{0.01}	ヘリウム	1.02	1.29
Fe _{0.1} Cu ₁ Nb ₃ Mo _{0.4} Si _{15.5} B _{0.2}	アルゴン	0.99	1.28
Fe _{0.1} Cu ₁ Nb ₃ Mn _{0.2} Si _{15.5} B _{0.5}	窒素	0.98	1.27
Fe _{0.1} Cu ₁ Nb ₃ Si _{15.5} B ₂ Ga _{0.3}	ヘリウム	1.02	1.29
Fe _{0.1} Cu ₁ Nb _{2.5} Cr _{0.5} Si _{13.5} B _{0.5}	窒素	1.01	1.33
Fe _{0.1} Cu ₁ Nb ₃ Si _{14.7} B ₂ Al _{0.01} Sn _{0.01}	窒素	1.00	1.27
Fe _{0.1} Cu ₁ Nb _{2.7} Mo _{0.5} Si ₁₅ B ₂ Co _{0.01}	窒素	1.05	1.32
Fe _{0.1} Cu _{1.5} Nb _{3.5} Si _{14.7} B ₂ Ge _{1.5}	窒素	1.03	1.26

【0016】露点が-10°Cと高い場合は μ_{re}/μ_r は1よりかなり大きく表面層除去により大きく透磁率が向上している。これに対して露点が-65°Cと低い場合はエッチングの影響はほとんどなく1に近い値である。これは露点が-65°Cと低い場合は磁気特性に影響を与える表面変

質層ができにくくことを示している。

【0017】

【発明の効果】本発明によれば、超微細な結晶粒組織を有する磁気特性に優れたナノ結晶合金の熱処理方法を提供することができるためその効果は著しいものがある。

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【手続補正書】

【提出日】平成13年3月7日(2001.3.7)

【手続補正1】

【補正対象書類名】明細書

【補正対象項目名】発明の名称

【補正方法】変更

【補正内容】

【発明の名称】ナノ結晶合金のナノ結晶化熱処理方法

【手続補正2】

【補正対象書類名】明細書

【補正対象項目名】特許請求の範囲

【補正方法】変更

【補正内容】

【特許請求の範囲】

【請求項1】 アモルファス合金を結晶化させて平均結晶粒径が30nm以下である結晶粒が組織の少なくとも一部を占めるナノ結晶合金を製造するナノ結晶化熱処理方法において、前記熱処理を露点が-30°C以下のガス雰囲気中で行なうことを特徴とするナノ結晶合金のナノ結晶化熱処理方法。

【請求項2】 前記露点が-60°C以下のガス雰囲気中であることを特徴とする請求項1に記載のナノ結晶合金のナノ結晶化熱処理方法。

【請求項3】 前記雰囲気ガスがアルゴンガス、ヘリウムガス、窒素ガスあるいはこれらの混合ガスであることを特徴とする請求項1または請求項2に記載のナノ結晶合金のナノ結晶化熱処理方法。

【請求項4】 ナノ結晶合金が一般式： $(Fe_{1-x}M_x)^{100-x-y-z-b}A_yM'^yM''^zX^z$ (原子%)で表され、式中MはC、Niから選ばれた少なくとも1種の元素を、AはCu、Auから選ばれた少なくとも1種の元素、M'はTi、V、Zr、Nb、Mo、H

f、TaおよびWから選ばれた少なくとも1種の元素、M''はCr、Mn、Al、Sn、Zn、Ag、In、白金属元素、Mg、Ca、Sr、Y、希土類元素、N、OおよびSから選ばれた少なくとも1種の元素、XはB、Si、C、Ge、GaおよびPから選ばれた少なくとも1種の元素を示し、a、x、y、zおよびbはそれぞれ0≤a≤0.5、0≤x≤10、0.1≤y≤20、0≤z≤20、2≤b≤30を満足する数で表される組成であることを特徴とする請求項1乃至請求項3のいずれかの項に記載のナノ結晶合金のナノ結晶化熱処理方法。

【手続補正3】

【補正対象書類名】明細書

【補正対象項目名】0001

【補正方法】変更

【補正内容】

【0001】

【産業上の利用分野】本発明は、超微細な結晶粒組織を有する磁性部品等に使用されるナノ結晶合金のナノ結晶化熱処理方法に関する。

【手続補正4】

【補正対象書類名】明細書

【補正対象項目名】0003

【補正方法】変更

【補正内容】

【0003】

【発明が解決しようとする課題】ナノ結晶合金のナノ結晶化熱処理は通常窒素ガスやアルゴン等の不活性ガス中で行われるが使用するガスによりしばしば磁気特性に劣下が生ずる場合があり問題となっていた。本発明の目的は均一で超微細な結晶粒組織を有する磁気特性に優れたナノ結晶合金のナノ結晶化熱処理方法を提供することである。

【手続補正5】

【補正対象書類名】明細書

【補正対象項目名】0004

【補正方法】変更

【補正内容】

【0004】

【課題を解決するための手段】上記問題点を解決するために本発明者らは、鋭意検討を進めた結果、表面に酸化物や水酸化物等の変質層がある場合には、磁気特性が劣化したり、磁気特性自体のばらつきが大きいことを知見した。そこでこの変質層の発生を防ぐには、ナノ結晶化熱処理中のガス雰囲気中の水分量を制御する、つまりガス雰囲気中の露点を制御すれば良いことを知見し、本発明に想到した。すなわち、本発明は、アモルファス合金を結晶化させて平均結晶粒径が30nm以下である結晶粒が組織の少なくとも一部を占めるナノ結晶合金を製造するナノ結晶化熱処理方法において、ナノ結晶化熱処理を露点が-30°C以下のガス雰囲気中で行なうことを特徴とするナノ結晶合金のナノ結晶化熱処理方法である。露点を-30°C以下に限定したのは、露点が-30°Cを越えると合金表面変質層が増加し、透磁率等の磁気特性が劣下するためである。

【手続補正6】

【補正対象書類名】明細書

【補正対象項目名】0007

【補正方法】変更

【補正内容】

【0007】前述の結晶は主にbccFe相であり、Siを含む場合はbcc相中にはSiが固溶し規則格子を含む場合もある。また、Si以外の元素たとえばB, Al, Ge, Zr, Ga等を固溶している場合もある。前記結晶相以外の残部は主にアモルファス相であるが、実質的に結晶相だけからなる場合もある。本発明に係わる合金は、前記組成のアモルファス合金を単ロール法等の超急冷法により作製後、これを磁心の形状に加工し、露点が-30°C以下のガス雰囲気中で結晶化温度以上に昇温しナノ結晶化熱処理を行い、平均粒径30nm以下の超微結晶粒を形成することにより作製する。ナノ結晶化熱処理の際磁場を印加し磁場中熱処理を行っても良い。

【手続補正7】

【補正対象書類名】明細書

【補正対象項目名】0010

【補正方法】変更

【補正内容】

【0010】

【作用】本発明においてナノ結晶化熱処理時に使用する雰囲気ガスの露点を-30°C以下とすることにより磁気特性に影響を与える合金表面変質層を少なくでき、磁気特性の劣下を減少させることができる。

【手続補正8】

【補正対象書類名】明細書

【補正対象項目名】0011

【補正方法】変更

【補正内容】

【0011】

【実施例】以下本発明を実施例にしたがって説明するが本発明はこれらに限定されるものではない。

(実施例1) 原子%でCu 1%, Nb 3%, Si 15.4%, B 6.5% 残部実質的にFeからなる合金溶湯を単ロール法により急冷し、幅5mm厚さ18μmのアモルファス合金を得た。このアモルファス合金を外径20mm、内径12mmに巻回し、トロイダル磁心を作製した。作製した磁心を表1に示す露点が異なる窒素ガス雰囲気の550°Cに保った熱処理炉に挿入し、30分保持後炉から取り出し空冷した。ナノ結晶化熱処理後の合金は粒径約12nmのbcc結晶が組織のほとんどを占めていた。1kHzにおける比透磁率 μ_r を表1に示す。表1から分るように露点が-30°C以下になると μ_r が著しく向上することが分る。特に-60°C以下で高い透磁率が得られた。

【手続補正9】

【補正対象書類名】明細書

【補正対象項目名】0013

【補正方法】変更

【補正内容】

【0013】(実施例2) 表2に示す組成の合金溶湯を単ロール法により急冷し、幅25mm厚さ16μmのアモルファス合金を得た。このアモルファス合金を外径20mm、内径12mmに巻回し、トロイダル磁心を作製した。作製した各磁心をそれぞれ露点が-65°Cと-10°Cの表2に示す雰囲気ガスの550°Cに保った熱処理炉に挿入し30分保持後炉から取り出し空冷した。ナノ結晶化熱処理後の合金は粒径約12nmのbcc結晶が組織のほとんどを占めていた。得られた磁気特性を表2に示す。表2から分るように露点が-65°Cと低い方が透磁率が向上することが分る。更に同様の条件で長さ200mmの前記合金をナノ結晶化熱処理し、1kHzにおける比透磁率 μ_r を測定した。測定後の試料表面をエッティングにより除去し、再度比透磁率 $\mu_{r,2}$ を測定した。エッティング前の比透磁率とエッティング後の比透磁率の比 $\mu_{r,2}/\mu_r$ を表3に示す。

【手続補正10】

【補正対象書類名】明細書

【補正対象項目名】0017

【補正方法】変更

【補正内容】

【0017】

【発明の効果】本発明によれば、超微細な結晶粒組織を有する磁気特性に優れたナノ結晶合金のナノ結晶化熱処理方法を提供することができるためその効果は著しいものがある。

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CLAIMS

[Claim(s)]

[Claim 1] The heat treatment approach of the nano crystal alloy characterized by a dew-point performing said heat treatment in the gas ambient atmosphere below -30-degreeC in the heat treatment approach of manufacturing the nano crystal alloy with which an amorphous alloy is crystallized and the crystal grain whose diameter of average crystal grain is 30nm or less occupies some organizations [at least].

[Claim 2] The heat treatment approach of the nano crystal alloy according to claim 1 characterized by said dew-point being among the gas ambient atmosphere below -60-degreeC.

[Claim 3] The heat treatment approach of the nano crystal alloy according to claim 1 or 2 characterized by said controlled atmospheres being argon gas, gaseous helium, nitrogen gas, or these mixed gas.

[Claim 4] a nano crystal alloy -- general formula: $(Fe1-aMa) - 100-x-y-z-bAxM' yM' - zXb$ (atomic %) It is expressed. At least one sort of elements with which A was chosen from Cu and Au in at least one sort of elements with which it was chosen out of Co and nickel by the inside M of a formula, At least one sort of elements with which M' was chosen from Ti, V, Zr, Nb, Mo, Hf, Ta, and W, At least one sort of elements with which M' was chosen from Cr, Mn, aluminum, Sn, Zn, Ag, In, platinum group elements, Mg, calcium, Sr and Y, rare earth elements, and N, O and S, X shows at least one sort of elements chosen from B, Si, C, germanium, Ga, and P. ax, y, z, and b are the heat treatment approach of a nano crystal alloy given in the term of either claim 1 characterized by being the presentation expressed with $0 \leq a \leq 0.5$, $0 \leq x \leq 10$, $0.1 \leq y \leq 20$, $0 \leq z \leq 20$, and the number with which are satisfied of $2 \leq b \leq 30$ thru/or claim 3, respectively.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the heat treatment approach of the nano crystal alloy used for the magnetic components which have the overly detailed grain structure.

[0002]

[Description of the Prior Art] The nano crystal alloy is used for the core of KOMOMMO-DOCHO-KUKOIRU, a high frequency transformer, a leakage alarm, a pulse transformer, etc. in order to show outstanding soft magnetic characteristics. As for the typical presentation system, the alloy system of a publication etc. is known by JP, 4-4393, B and JP, 1-242755, A. These nano crystal alloys are produced by microcrystal-izing this by heat treatment, after usually quenching from the liquid phase or a gaseous phase and considering as an amorphous alloy. It quenches from the liquid phase and the single rolling method, the congruence rolling method, the centrifugal quenching method, the spinning method in revolution liquid, the atomizing method, the cavitation method, etc. are learned as an approach of making it into an amorphous alloy. Moreover, it quenches from a gaseous phase and a spatter, vacuum deposition, the ion plating method, etc. are learned as an approach of making it into an amorphous alloy. A nano crystal alloy is what microcrystal-ized the amorphous alloy produced by these approaches, and does not almost have thermal instability nature which is seen by the amorphous alloy, and it is known that soft magnetic characteristics excellent in high saturation magnetic flux density and low magnetostriction are shown. Furthermore, a nano crystal alloy has small aging and excelling also in the temperature characteristic is known.

[0003]

[Problem(s) to be Solved by the Invention] Degradation may often arise

in magnetic properties by the gas used although heat treatment of a nano crystal alloy is usually performed in inert gas, such as nitrogen gas and an argon, and it had become a problem. The object of this invention is offering the heat treatment approach of a nano crystal alloy excellent in the magnetic properties which have the uniform and overly detailed grain structure.

[0004]

[Means for Solving the Problem] In order to solve the above-mentioned trouble, magnetic properties deteriorated and this invention persons did the knowledge of dispersion in the magnetic properties itself being large, when deterioration layers, such as an oxide and a hydroxide, were shown in a front face, as a result of advancing examination wholeheartedly. Then, in order to have prevented generating of this deterioration layer, the knowledge of what is necessary being to control the moisture content in the gas ambient atmosphere under heat treatment, that is, just to control the dew-point in a gas ambient atmosphere was carried out, and it hit on an idea to this invention. That is, this invention is the heat treatment approach of the nano crystal alloy characterized by a dew-point heat-treating in the gas ambient atmosphere below -30-degreeC in the heat treatment approach of manufacturing the nano crystal alloy with which an amorphous alloy is crystallized and the crystal grain whose diameter of average crystal grain is 30nm or less occupies some organizations [at least]. The dew-point was limited to below -30-degreeC for an alloy surface deterioration layer increasing, if a dew-point exceeds -30-degreeC, and magnetic properties, such as permeability, deteriorating.

[0005] When especially a dew-point is among the gas ambient atmosphere below -60-degreeC, magnetic properties improve further, and a more desirable result is obtained. The moisture content of 337.7 mg/m³ and -60 degrees of dew-points C are equivalent to the moisture content of 10.93 mg/m³ for dew-point-30-degreeC. When argon gas, gaseous helium, inactive nitrogen gas, or these inactive mixed gas is used as a controlled atmosphere, outstanding magnetic properties are acquired and the effectiveness of this invention is remarkable.

[0006] a nano crystal soft magnetism alloy -- especially -- general formula: (Fe_{1-a}M_a) -- 100-x-y-z-bAxM'yM' -- 'zXb (atomic %) It is expressed. At least one sort of elements with which A was chosen from Cu and Au in at least one sort of elements with which it was chosen out of Co and nickel by the inside M of a formula, At least one sort of elements with which M' was chosen from Ti, V, Zr, Nb, Mo, Hf, Ta, and W, At least one sort of elements with which M' was chosen from Cr, Mn,

aluminum, Sn, Zn, Ag, In, platinum group elements, Mg, calcium, Sr and Y, rare earth elements, and N, O and S, X shows at least one sort of elements chosen from B, Si, C, germanium, Ga, and P. The soft magnetic characteristics which were excellent in the presentation expressed with the number with which ax , y , z , and b are satisfied of $0 \leq a \leq 0.5$, $0 \leq x \leq 10$, $0.1 \leq y \leq 20$, $0 \leq z \leq 20$, and $2 \leq b \leq 30$, respectively are obtained. [0007] The above-mentioned crystal is mainly a bccFe phase, and when Si is included, Si may dissolve in a bcc phase and it may contain a superlattice. Moreover, it may be dissolving in elements other than Si, for example, B, aluminum, germanium, Zr, Ga, etc. Although the remainders other than said crystal phase are mainly amorphous phases, they may consist only of a crystal phase substantially. The amorphous alloy of said presentation is processed after production, and it processes this into the configuration of a core with super-quenching methods, such as the single rolling method, and the alloy concerning this invention heat-treats by a dew-point carrying out temperature up in the gas ambient atmosphere below -30-degreeC beyond crystallization temperature, and produces it by forming a super-microcrystal grain with a mean particle diameter of 30nm or less. A magnetic field may be impressed in the case of heat treatment, and heat treatment among a magnetic field may be performed.

[0008] Since heat dissipation of the heat by crystallization from a core front face to generate becomes good and unusual lifting of core temperature can be suppressed low, moving the controlled atmosphere in a furnace compulsorily can obtain a more desirable result. While introducing a controlled atmosphere in a furnace from the outside of a furnace, the gas in a furnace can be discharged, and effectiveness with the same said of moving the controlled atmosphere in a furnace compulsorily can be acquired. Since making the controlled atmosphere in a furnace stir compulsorily, and moving it by a fan etc. can also improve heat dissipation from a core front face, the same effectiveness can be acquired.

[0009] As the difference of nano crystal alloy skin temperature and the laying temperature of a furnace becomes below 50-degreeC, also when a configuration becomes large by establishing the device in which the amount of furnace internal transmigration of a controlled atmosphere is adjusted, a response becomes possible easily. Especially when the difference of nano crystal alloy skin temperature and the laying temperature of a furnace is below 10-degreeC, a result with them is obtained. [degradation of a property and small dispersion of a property, and] [desirable]

[0010]

[Function] By making below into -30-degreeC the dew-point of the controlled atmosphere used in this invention at the time of heat treatment, the alloy surface deterioration layer which affects magnetic properties can be lessened, and the degradation of magnetic properties can be decreased.

[0011]

[Example] Although this invention is explained according to an example below, this invention is not limited to these.

(Example 1) The alloy molten metal which becomes Cu 1%, Nb 3%, Si 15.4%, and a B 6.5% remainder real target from Fe by atomic % was quenched by the single rolling method, and the with a width-of-face thickness [18-micrometer thickness of 5mm] amorphous alloy was obtained. Winding and a toroidal core were produced for this amorphous alloy in the outer diameter of 20mm, and bore of 12mm. It inserted in the heat treating furnace maintained at 550-degreeC of nitrogen-gas-atmosphere mind that the dew-points which show the produced core in a table 1 differ, and ejection air cooling was carried out from the after [30 minute maintenance] furnace. As for the alloy after heat treatment, the bcc crystal with a particle size of about 12nm occupied most organizations. the ratio in 1kHz -- initial permeability μ_r is shown in a table 1. As shown in a table 1, when a dew-point becomes below -30-degreeC, it turns out that μ_r improves remarkably. High permeability was especially obtained below by -60-degreeC.

[0012]

[A table 1]

露点 (°C)	μ_r
-10	63200
-20	73000
-30	90100
-50	95500
-60	97200
-70	98300
-80	98500

[0013] (Example 2) The alloy molten metal of the presentation shown in a table 2 was quenched by the single rolling method, and the with a width-of-face thickness [16-micrometer thickness of 25mm] amorphous alloy was obtained. Winding and a toroidal core were produced for this

amorphous alloy in the outer diameter of 20mm, and bore of 12mm. The dew-point inserted each produced core in the heat treating furnace maintained at 550-degreeC of the controlled atmosphere shown in the table 2 of -65-degreeC and -10-degreeC, and carried out ejection air cooling from the after [30 minute maintenance] furnace, respectively. As for the alloy after heat treatment, the bcc crystal with a particle size of about 12nm occupied most organizations. The acquired magnetic properties are shown in a table 2. It turns out that the one of permeability where a dew-point is as low as -65-degreeC improves as shown in a table 2. Furthermore, said alloy with a die length of 200mm was heat-treated on the same conditions, and relative permeability μ_r in 1kHz was measured. Etching removed the sample front face after measurement, and relative permeability μ_r was measured again. the ratio of the relative permeability before etching, and the relative permeability after etching -- μ_r / μ_r is shown in a table 3.

[0014]

[A table 2]

組成 (at%)	雰囲気ガス	μ_r [露点-65°C]	μ_r [露点-10°C]
Fe _{64.1} Cu ₁ Nb ₃ Si _{14.5} B _{8.2}	アルゴン	97200	62100
Fe _{64.1} Cu ₁ Nb ₃ Ti _{0.7} Si ₁₄ B ₈	窒素	98100	71500
Fe _{64.1} Cu ₁ Nb ₃ Si _{14.7} B ₇ Sn _{0.01}	ヘリウム	96200	74800
Fe _{64.1} Cu ₁ Nb ₃ Mo _{0.4} Si _{15.5} B _{8.2}	アルゴン	93800	73200
Fe _{64.1} Cu ₁ Nb ₃ Mn _{0.2} Si _{15.5} B _{8.5}	窒素	91200	71800
Fe _{64.1} Au _{0.9} Nb ₃ Si _{15.5} B ₈ Ga _{0.5}	ヘリウム	89600	69500
Fe _{64.1} Cu ₁ Nb _{2.5} Cr _{0.5} Si _{15.5} B _{8.5}	窒素	88700	66800
Fe _{64.1} Cu ₁ Nb ₃ Si ₁₄ B ₈ Al _{0.01} Sn _{0.06}	窒素	91200	71900
Fe _{64.1} Cu ₁ Nb _{2.7} Mo _{0.5} Si ₁₅ B ₈ Co _{0.01}	窒素	87300	65900
Fe _{64.1} Cu _{1.5} Nb _{3.5} Si _{14.7} B ₈ Ge _{1.5}	窒素	91300	72300

[0015]

[A table 3]

組成 (at%)	雰囲気ガス	μ_{re}/μ_r [露点-65°C]	μ_{re}/μ_r [露点-10°C]
Fe _{6.1} Cu ₁ Nb ₃ Si _{14.5} B _{9.2}	アルゴン	1.02	1.53
Fe _{6.1} Cu ₁ Nb ₃ Ti _{0.7} Si ₁₄ B ₉	窒素	1.09	1.36
Fe _{6.1} Cu ₁ Nb ₃ Si _{14.7} B ₇ Sn _{0.01}	ヘリウム	1.02	1.29
Fe _{6.1} Cu ₁ Nb ₃ Mo _{0.4} Si _{15.5} B _{9.2}	アルゴン	0.99	1.28
Fe _{6.1} Cu ₁ Nb ₃ Mn _{0.2} Si _{15.5} B _{8.5}	窒素	0.98	1.27
Fe _{6.1} Au _{0.6} Nb ₃ Si _{15.5} B ₉ Ga _{0.3}	ヘリウム	1.02	1.29
Fe _{6.1} Cu ₁ Nb _{2.5} Cr _{0.5} Si _{13.5} B _{8.5}	窒素	1.01	1.33
Fe _{6.1} Cu ₁ Nb ₃ Si ₁₄ B ₈ Al _{0.01} Sn _{0.06}	窒素	1.00	1.27
Fe _{6.1} Cu ₁ Nb _{2.7} Mo _{0.5} Si ₁₅ B ₈ Co _{0.01}	窒素	1.05	1.32
Fe _{6.1} Cu _{1.6} Nb _{8.5} Si _{14.7} B ₈ Ge _{1.5}	窒素	1.03	1.26

[0016] When a dew-point is as high as -10-degreeC, permeability of μ_{re}/μ_r is improving greatly by surface-layer clearance quite more greatly than 1. On the other hand, when a dew-point is as low as -65-degreeC, there is almost no effect of etching and it is a value near 1. When this has a dew-point as low as -65-degreeC, it is shown that the surface deterioration layer which affects magnetic properties cannot be made easily.

[0017]

[Effect of the Invention] According to this invention, since it can ** if the heat treatment approach of a nano crystal alloy excellent in the magnetic properties which have the overly detailed grain structure is offered, a remarkable thing has the effectiveness.

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CLAIMS

[Claim(s)]

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[Claim 2] The heat treatment approach of the nano crystal alloy according to claim 1 characterized by said dew-point being among the gas ambient atmosphere below -60-degreeC.

[Claim 3] The heat treatment approach of the nano crystal alloy according to claim 1 or 2 characterized by said controlled atmospheres being argon gas, gaseous helium, nitrogen gas, or these mixed gas.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the heat treatment approach of the nano crystal alloy used for the magnetic components which have the overly detailed grain structure.

[0002]

[Description of the Prior Art] The nano crystal alloy is used for the core of KOMOMMO-DOCHO-KUKOIRU, a high frequency transformer, a leakage alarm, a pulse transformer, etc. in order to show outstanding soft magnetic characteristics. As for the typical presentation system, the alloy system of a publication etc. is known by JP, 4-4393, B and JP, 1-242755, A. These nano crystal alloys are produced by microcrystal-izing this by heat treatment, after usually quenching from the liquid phase or a gaseous phase and considering as an amorphous alloy. It quenches from the liquid phase and the single rolling method, the congruence rolling method, the centrifugal quenching method, the spinning method in revolution liquid, the atomizing method, the cavitation method, etc. are learned as an approach of making it into an amorphous alloy. Moreover, it quenches from a gaseous phase and a spatter, vacuum deposition, the ion plating method, etc. are learned as an approach of making it into an amorphous alloy. A nano crystal alloy is what microcrystal-ized the amorphous alloy produced by these approaches, and does not almost have thermal instability nature which is seen by the amorphous alloy, and it is known that soft magnetic characteristics excellent in high saturation magnetic flux density and low magnetostriction are shown. Furthermore, a nano crystal alloy has small aging and excelling also in the temperature characteristic is known.

[0003]

[Problem(s) to be Solved by the Invention] Degradation may often arise

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in magnetic properties by the gas used although heat treatment of a nano crystal alloy is usually performed in inert gas, such as nitrogen gas and an argon, and it had become a problem. The object of this invention is offering the heat treatment approach of a nano crystal alloy excellent in the magnetic properties which have the uniform and overly detailed grain structure.

[0004]

[Means for Solving the Problem] In order to solve the above-mentioned trouble, magnetic properties deteriorated and this invention persons did the knowledge of dispersion in the magnetic properties itself being large, when deterioration layers, such as an oxide and a hydroxide, were shown in a front face, as a result of advancing examination wholeheartedly. Then, in order to have prevented generating of this deterioration layer, the knowledge of what is necessary being to control the moisture content in the gas ambient atmosphere under heat treatment, that is, just to control the dew-point in a gas ambient atmosphere was carried out, and it hit on an idea to this invention. That is, this invention is the heat treatment approach of the nano crystal alloy characterized by a dew-point heat-treating in the gas ambient atmosphere below -30-degreeC in the heat treatment approach of manufacturing the nano crystal alloy with which an amorphous alloy is crystallized and the crystal grain whose diameter of average crystal grain is 30nm or less occupies some organizations [at least]. The dew-point was limited to below -30-degreeC for an alloy surface deterioration layer increasing, if a dew-point exceeds -30-degreeC, and magnetic properties, such as permeability, deteriorating.

[0005] When especially a dew-point is among the gas ambient atmosphere below -60-degreeC, magnetic properties improve further, and a more desirable result is obtained. The moisture content of 337. 7 mg/m³ and -60 degrees of dew-points C are equivalent to the moisture content of 10. 93 mg/m³ for dew-point-30-degreeC. When argon gas, gaseous helium, inactive nitrogen gas, or these inactive mixed gas is used as a controlled atmosphere, outstanding magnetic properties are acquired and the effectiveness of this invention is remarkable.

[0006] a nano crystal soft magnetism alloy -- especially -- general formula: (Fe_{1-a}Ma) -- 100-x-y-z-bAxM' yM' -- 'zXb (atomic %) It is expressed. At least one sort of elements with which A was chosen from Cu and Au in at least one sort of elements with which it was chosen out of Co and nickel by the inside M of a formula, At least one sort of elements with which M' was chosen from Ti, V, Zr, Nb, Mo, Hf, Ta, and W, At least one sort of elements with which M'' was chosen from Cr, Mn,

aluminum, Sn, Zn, Ag, In, platinum group elements, Mg, calcium, Sr and Y, rare earth elements, and N, O and S, X shows at least one sort of elements chosen from B, Si, C, germanium, Ga, and P. The soft magnetic characteristics which were excellent in the presentation expressed with the number with which a_x , y , z , and b are satisfied of $0 \leq a \leq 0.5$, $0 \leq x \leq 10$, $0.1 \leq y \leq 20$, $0 \leq z \leq 20$, and $2 \leq b \leq 30$, respectively are obtained.

[0007] The above-mentioned crystal is mainly a bccFe phase, and when Si is included, Si may dissolve in a bcc phase and it may contain a superlattice. Moreover, it may be dissolving in elements other than Si, for example, B, aluminum, germanium, Zr, Ga, etc. Although the remainders other than said crystal phase are mainly amorphous phases, they may consist only of a crystal phase substantially. The amorphous alloy of said presentation is processed after production, and it processes this into the configuration of a core with super-quenching methods, such as the single rolling method, and the alloy concerning this invention heat-treats by a dew-point carrying out temperature up in the gas ambient atmosphere below -30-degreeC beyond crystallization temperature, and produces it by forming a super-microcrystal grain with a mean particle diameter of 30nm or less. A magnetic field may be impressed in the case of heat treatment, and heat treatment among a magnetic field may be performed.

[0008] Since heat dissipation of the heat by crystallization from a core front face to generate becomes good and unusual lifting of core temperature can be suppressed low, moving the controlled atmosphere in a furnace compulsorily can obtain a more desirable result. While introducing a controlled atmosphere in a furnace from the outside of a furnace, the gas in a furnace can be discharged, and effectiveness with the same said of moving the controlled atmosphere in a furnace compulsorily can be acquired. Since making the controlled atmosphere in a furnace stir compulsorily, and moving it by a fan etc. can also improve heat dissipation from a core front face, the same effectiveness can be acquired.

[0009] As the difference of nano crystal alloy skin temperature and the laying temperature of a furnace becomes below 50-degreeC, also when a configuration becomes large by establishing the device in which the amount of furnace internal transmigration of a controlled atmosphere is adjusted, a response becomes possible easily. Especially when the difference of nano crystal alloy skin temperature and the laying temperature of a furnace is below 10-degreeC, a result with them is obtained. [degradation of a property and small dispersion of a property, and] [desirable]

[0010]

[Function] By making below into -30-degreeC the dew-point of the controlled atmosphere used in this invention at the time of heat treatment, the alloy surface deterioration layer which affects magnetic properties can be lessened, and the degradation of magnetic properties can be decreased.

[0011]

[Example] Although this invention is explained according to an example below, this invention is not limited to these.

(Example 1) The alloy molten metal which becomes Cu 1%, Nb 3%, Si 15.4%, and a B 6.5% remainder real target from Fe by atomic % was quenched by the single rolling method, and the with a width-of-face thickness [18-micrometer thickness of 5mm] amorphous alloy was obtained. Winding and a toroidal core were produced for this amorphous alloy in the outer diameter of 20mm, and bore of 12mm. It inserted in the heat treating furnace maintained at 550-degreeC of nitrogen-gas-atmosphere mind that the dew-points which show the produced core in a table 1 differ, and ejection air cooling was carried out from the after [30 minute maintenance] furnace. As for the alloy after heat treatment, the bcc crystal with a particle size of about 12nm occupied most organizations. the ratio in 1kHz -- initial permeability mur is shown in a table 1. As shown in a table 1, when a dew-point becomes below -30-degreeC, it turns out that mur improves remarkably. High permeability was especially obtained below by -60-degreeC.

[0012]

[A table 1]

露点 (°C)	μr
-10	63200
-20	73000
-30	90100
-50	95500
-60	97200
-70	98900
-80	98500

[0013] (Example 2) The alloy molten metal of the presentation shown in a table 2 was quenched by the single rolling method, and the with a width-of-face thickness [16-micrometer thickness of 25mm] amorphous alloy was obtained. Winding and a toroidal core were produced for this

amorphous alloy in the outer diameter of 20mm, and bore of 12mm. The dew-point inserted each produced core in the heat treating furnace maintained at 550-degreeC of the controlled atmosphere shown in the table 2 of -65-degreeC and -10-degreeC, and carried out ejection air cooling from the after [30 minute maintenance] furnace, respectively. As for the alloy after heat treatment, the bcc crystal with a particle size of about 12nm occupied most organizations. The acquired magnetic properties are shown in a table 2. It turns out that the one of permeability where a dew-point is as low as -65-degreeC improves as shown in a table 2. Furthermore, said alloy with a die length of 200mm was heat-treated on the same conditions, and relative permeability μ_r in 1kHz was measured. Etching removed the sample front face after measurement, and relative permeability μ_r was measured again. the ratio of the relative permeability before etching, and the relative permeability after etching -- μ_r / μ_r is shown in a table 3.

[0014]

[A table 2]

組成 (at%)	雰囲気ガス	μ_r [露点-65°C]	μ_r [露点-10°C]
Fe _{61.1} Cu ₁ Nb ₃ Si _{14.5} B _{8.2}	アルゴン	97200	62100
Fe _{61.1} Cu ₁ Nb ₃ Ti _{0.7} Si ₁₄ B ₈	窒素	98100	71500
Fe _{61.1} Cu ₁ Nb ₃ Si _{14.7} B ₇ Sn _{0.01}	ヘリウム	96200	74800
Fe _{61.1} Cu ₁ Nb ₃ Mo _{0.4} Si _{15.5} B _{8.2}	アルゴン	93800	73200
Fe _{61.1} Cu ₁ Nb ₃ Mn _{0.2} Si _{16.5} B _{8.5}	窒素	91200	71800
Fe _{61.1} Au _{0.9} Nb ₃ Si _{16.5} B ₈ Ga _{0.5}	ヘリウム	89600	69500
Fe _{61.1} Cu ₁ Nb _{2.5} Cr _{0.5} Si _{15.5} B _{8.5}	窒素	88700	66800
Fe _{61.1} Cu ₁ Nb ₃ Si _{14.8} B ₈ Al _{0.01} Sn _{0.01}	窒素	91200	71900
Fe _{61.1} Cu ₁ Nb _{2.7} Mo _{0.5} Si ₁₅ B ₈ Co _{0.01}	窒素	87300	65900
Fe _{61.1} Cu _{1.6} Nb _{3.5} Si _{14.7} B ₈ Ge _{1.6}	窒素	91300	72300

[0015]

[A table 3]

組成 (at%)	雰囲気ガス	μ_{re}/μ_r [露点-65°C]	μ_{re}/μ_r [露点-10°C]
Fe _{6.1} Cu ₁ Nb ₃ Si _{14.6} B _{8.2}	アルゴン	1.02	1.53
Fe _{6.1} Cu ₁ Nb ₃ Ti _{0.7} Si ₁₄ B ₉	窒素	1.09	1.36
Fe _{6.1} Cu ₁ Nb ₃ Si _{14.7} B ₇ Sn _{0.01}	ヘリウム	1.02	1.29
Fe _{6.1} Cu ₁ Nb ₃ Mo _{0.4} Si _{16.5} B _{8.2}	アルゴン	0.99	1.28
Fe _{6.1} Cu ₁ Nb ₃ Mn _{0.2} Si _{15.6} B _{8.6}	窒素	0.98	1.27
Fe _{6.1} Au _{0.9} Nb ₃ Si _{15.6} B ₈ Ga _{0.5}	ヘリウム	1.02	1.29
Fe _{6.1} Cu ₁ Nb _{2.5} Cr _{0.5} Si _{13.6} B _{8.6}	窒素	1.01	1.33
Fe _{6.1} Cu ₁ Nb ₃ Si ₁₄ B ₈ Al _{0.01} Sn _{0.00}	窒素	1.00	1.27
Fe _{6.1} Cu ₁ Nb _{2.7} Mo _{0.5} Si ₁₅ B ₈ Co _{0.01}	窒素	1.05	1.32
Fe _{6.1} Cu _{1.6} Nb _{8.6} Si _{14.7} B ₈ Ge _{1.6}	窒素	1.03	1.26

[0016] When a dew-point is as high as -10-degreeC, permeability of μ_{re}/μ_r is improving greatly by surface-layer clearance quite more greatly than 1. On the other hand, when a dew-point is as low as -65-degreeC, there is almost no effect of etching and it is a value near 1. When this has a dew-point as low as -65-degreeC, it is shown that the surface deterioration layer which affects magnetic properties cannot be made easily.

[0017]

[Effect of the Invention] According to this invention, since it can ** if the heat treatment approach of a nano crystal alloy excellent in the magnetic properties which have the overly detailed grain structure is offered, a remarkable thing has the effectiveness.

[Translation done.]